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Research Article

Effect of Microwave Treatment on the Profile of Volatile Compounds and Characteristics of White Pepper (*Piper nigrum* L.) Essential Oil

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ABSTRACT

*Microwave treatment on white pepper was conducted to damage cell tissue to facilitate the distillation and increase the yield of essential oils. The research objective was to determine the effect of pepper varieties and microwave treatment on the profiles of volatile compounds and the characteristics of white pepper essential oils. The research was conducted with 50 grams of white pepper placed into a 15 cm diameter petri dish and put in a microwave oven at the power of 600 watts for 90 seconds then white pepper milled by hammer mill. Essential oils were obtained using the water distillation method then were tested for their characteristics and compounds using Gas chromatography-Mass Spectrometry (GC-MS). White pepper was analyzed using the water content, piperine content, and its cell tissue microstructure was analyzed using Scanning Electron Microscope (SEM). The results showed that different pepper as accession produced different profiles of volatile compounds, characteristics, and antioxidant activity (p<0.05). The number of volatile compounds of white pepper essential oils identified by GC-MS was 42, where the largest compound was β-caryophyllene (50.51%). Microwave treatment damaged the cell wall of white pepper, lowered water content (17.43%), increased piperine content (10.57%) and essential oil yield (25%), and changed the profiles of volatile compounds of essential oils of Lampung daun lebar accession. Color became bluer (b*value rises by 201.99%), increased specific gravity (1.27%) and antioxidant activity (7.4%), decreased solubility in 95% ethanol (17.95%) and acid number (20.8%) while the refractive index was not affected by pepper accessions and microwave treatment.*

Keywords:* *Essential oils; Microwave treatment; Volatile compounds; White pepper.

1. Introduction

Pepper is one of Indonesia's traditional mainstay export commodities, which is obtained from the plant fruit *Piper nigrum* Linn. Indonesia is the third-largest pepper exporting country after Vietnam (Sudjarmoko *et al.* 2015). Pepper is known as *piper*, *merica*, or *sahang*. Pepper fruit contains essential oils, alkaloids, resins, proteins, cellulose, pentosan, starch, minerals, and others. Pepper aroma is influenced by volatile oil and its volatile compounds. Piperine is the main component that causes edible flavor in pepper (Nitin *et al.* 2012). Pepper essential oils have potential for biopesticide agent (Abdullah *et al.* 2020), antibacterial agent (Abdallah & Abdalla 2018), analgesic (Costa *et al.* 2016), and natural insecticide (Chaubey, 2017), as well as antioxidant activity (Gülçin, 2005; Abd El Mageed *et al.* 2011).

The essential oil yield of Indonesia's white pepper ranges between 2-3% (Kusmiadi *et al.* 2017; Syakir *et al.* 2017; Putri *et al.* 2018). The yield obtained can be influenced by the distillation method used, comparative research of the profiles of volatile essential oils from water distillation with the simultaneous distillation extraction (SDE) method (Chen *et al.* 2011), the difference between the volatile compounds of essential oils resulting from hydrodistillation and Microwave-Assisted Extraction (MAE) (Rmili *et al*. 2014) and advanced method with solvent-free simultaneous ultrasonic-microwave assisted extraction (Wang *et al.* 2018). Principal Component Analysis (PCA) analyzes the profile of volatile white pepper compounds from different grinding method and accession form then classifies them into different quadrants (Liu *et al.* 2013; Fan *et al.* 2020)

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White pepper essential oil is inside the cell vacuole coated with a hard cell wall structure. Dielectric heating that occurs due to the impact of microwave can degrade cellulose compounds in the white pepper cell wall so that the permeability of the cell wall decreases (Raman & Gaikar 2002). In this condition, the yield obtained increases because during the distillation process, essential oils can come out more easily. Microwave treatment at 400 watts of power for 4 minutes affected both cell walls and the membrane cell of hazelnut seed and the largest extraction yield of oils was obtained (Uquiche *et al.* 2008). The yield of white pepper essential oil extracted by microwave assisted was 3.7% and higher than assisted by ultrasonic which was 3.4% (Wang *et al.* 2018). Black pepper which was given a microwave treatment at 663 watts of power had a higher volatile oil content than that of without microwave pre-treatment (Jeevitha *et al.* 2016). The use of microwave pre-treatment at 450 watts of power for 2 minutes can damage the microstructure of crushed nutmeg seed tissue so that it can increase the essential oil yield (Lumbessy 2016).

Different genotypes/accessions of pepper, the levels of essential oils are also different (Liu *et al.* 2013; Aziz *et al.* 2018). The differences in volatile compounds which make up essential oils were reported (Hao *et al.* 2018; Dosoky *et al.* 2019; van Ruth *et al.* 2019). Lampung daun lebar, Jambi, and Merapin jumbo are local accessions of pepper that are widely cultivated in Kepulauan Bangka Belitung Province (Prayoga *et al.* 2020). Therefore, in this study, it is suspected that there are differences in the profile of volatile compounds, characteristics, and antioxidant activity of these 3 (three) accessions.

This study aims to analyze the profiles of volatile compounds, and the characteristics (essential oil yield, color, specific gravity, refractive index, solubility in 95% ethanol, acid number, and antioxidant activity) of white pepper essential oil (*Piper nigrum* L.) accessions of Lampung daun lebar, Jambi, and Merapin jumbo which were given microwave treatment.

2. Materials and Methods

White pepper was processed from accessions of Lampung daun lebar, Jambi, and Merapin jumbo. Pepper fruit was harvested from 3-5 years old pepper plants in the 2017 harvest season. White pepper with water content below 14% of each variety was packed in 1 kg *low-density polyethylene* (LDPE) plastic coated with *aluminum foil* then stored in a closed plastic container and protected from heat and direct sunlight until used.

The main equipment in this research were the Samsung microwave oven type ME731K (power source 230 V - 50Hz, power consumption 1150 watts, power output 100-800 watts, operating frequency 2450 MHz, volume 20 L), SEM JSM-500, GC-MS Shimadzu QP 2010 SE with column TG-5MS, pycnometer, refractometer, UV-Vis spectrophotometry, Konica Minolta chromameter, a laboratory-scale distillation set consisting of a 2000 ml erlenmeyer, thermometer, condenser, electric stove, distillate container bottle, and aerator.

Microwave treatment

White pepper accessions of Lampung daun lebar (L), Jambi (J), and Merapin jumbo (M) (± 50 gram) were taken randomly from the storages The microwave treatment method referred to the most optimized result of Syafrudin (2016), the samples were placed in a petri dish with a diameter of 15 cm and then put in a microwave oven with a treatment time of 90 seconds at 600 watts of power. After that, is the samples were rested for ± 30 minutes so that the samples reach room temperature. The energy intensity to be received by the material was calculated according to the formula in Chávez-Reyes et al. (2013) which is equal to 1.08 Kj g-1. White pepper was mashed using a hammer mill and sieved to obtain 0.5 mm particle size. Eventually, there were 3 (three) accessions without microwave treatment which were Lampung daun lebar without treatment (LTP), Jambi without treatment (JTP) and Merapin jumbo without treatment (MTP), and 3 accessions with microwave treatment which were Lampung with microwave treatment (LDP), Jambi with microwave treatment (JDP), and Merapin jumbo with microwave treatment (MDP).

Evaluation of moisture content, piperine content, and observation of microstructure of white pepper cells

Water content analysis and piperine content analysis referred to the method of SNI 0004: 2013 white pepper (National Standardization Agency 2013). Microstructure observation of white pepper cell tissue was carried out on white pepper powder without treatment and with microwave treatment using *Scanning Electron Microscopy* (SEM). The observation method using SEM referred to the LPPT UGM where the magnification scale used starts from 3,000 to 15,000 times.

GC-MS analysis

GC MS analysis method referred to the UII Integrated Laboratory. Essential oil of white pepper as much as 1 μL was injected using a split method with a ratio of 1:80. GC-MS was operated using a column of TG-5MS 30 m long, 0.25 mm in diameter, and 0.25 μm thickness with the oven regulated between 60-200 ˚C with a temperature increase rate of 10 ˚C minute-1. The helium carrier gas was pressurized to 12.7 kPa and the total rate was 115,4 mL min-1. Library uses WILEY7.LIB.

White pepper essential oil characteristics

Essential oils were obtained and the yield was calculated using water distillation method SNI 0004:2013, color (CIE *L**, *a**, and *b**), specific gravity (ISO 279:1998 E), refractive index (ISO 280:1998 E), solubility in 95% ethanol (ISO 875:1999 E), and acid number (ISO 660:1996).

Antioxidant Activities

Antioxidant activity measurement referred to Gülçin (2005) method. 4mg DPPH was dissolved in 96% ethanol to 100 ml to obtain 0.004% solution (40 ppm). The arrays were maintained at low temperatures and protected from light. DPPH solution was added with ethanol 96%, with a ratio of 3:2 (v / v), for blank solution 96% ethanol was used, homogenized with vortex, and absorbance was observed at a range of wavelengths (λ) 517 nm. The essential oil of 2 ml white pepper was added with DPPH solution (3 ml), homogenized with vortex incubated for 30 minutes then absorbed. Determination of percent of inhibition to DPPH compounds (*1,1-diphenyl-2-picrylhydrazyl*) uses the following formula:

$$\%\ inhibition = \frac{absorbance\ (control - object)}{Control\ absorbance}\ x\ 100\%$$

Data Analysis

The statistical test used variance analysis (ANOVA) with a significance level of 95%, if there is a significant difference, it is continued with the Duncan Multiple Range Test (DMRT). Data processing used SPSS 21 software. Test result data was displayed with standard deviation and significance letter notation

Multivariate analysis for the profiles of volatile compounds used Minitab Principal Component Analysis (PCA) 14. The results of the GC-MS chromatogram interpretation were arranged into profiles of volatile compounds and the percentage of peaks obtained by each compound. The data collected were analyzed using principal components analysis (PCA) to differentiate the odors of different white pepper samples (Liu, F. Zeng *et al.* 2013). It was inputted into Minitab14 *sheet* data and analyzed using *principal component analysis* to obtain *plot scores* and *loading plots* profile of white pepper volatile essential oil compounds from 3 (three) pepper accessions without and with microwave treatment.

3. Results

Water content, piperine content, and microstructure of white pepper cell tissue

The water and piperine content of white pepper are shown in Table 1. The results of the analysis showed that different pepper accession produced different levels of water and piperine content of white pepper. Microwave treatment was proven to be able to reduce white pepper water content. The water content of Lampung daun lebar accession decreased 15.29% (percentage from 13.67% to 11.58%), Jambi 15.26%, and Merapin jumbo 17.43%. In Lampung daun lebar and Jambi accessions, microwave treatment caused the decrease in piperine contents while in the Merapin jumbo, the piperine content obtained was higher than without microwave treatment. The interaction between the accessions of pepper and the microwave treatment affected the piperine content of white pepper. The effect of pepper accessions and the microwave treatment on the microstructure of white pepper cells is shown in Figure 1. Scanning Electron Microscope (SEM) images showed that there were changes in the white pepper cell wall structures.

Table 1. Water content (b/b) and Piperine content b/b (%) from 3 (three) accesions without and with microwave treatment

Sample	Water content b/b (%)	Piperine content b/b(%)
LTP	$13.67^c \pm 0.14$	$6.20^d \pm 0.24$
LDP	$11.58^a \pm 0.14$	$6.01^c \pm 0.54$
JTP	$13.37^b \pm 0.13$	$6.91^f \pm 0.01$
JDP	$11.33^a \pm 0.14$	$6.73^e \pm 0.02$
MTP	$13.83^c \pm 0.14$	$5.86^b \pm 0.15$
MDP	$11.42^a \pm 0.14$	$5.30^a \pm 0.02$

notes: different letters in the same column show significant differences ($p<0.05$)

Effect of pepper varieties and microwave treatment on white pepper essential oil yield

Different pepper accession produced different essential oil yield of white pepper (Figure 2). White pepper from all accessions with microwave treatment has a higher essential oil yield than without microwave treatment. The essential oil Lampung daun lebar increased 25% (percentage from 2.34% to 2,93%), Jambi 8.2%, and Merapin jumbo 17.24%. The highest yield of essential oils was in Jambi accession with microwave treatment.

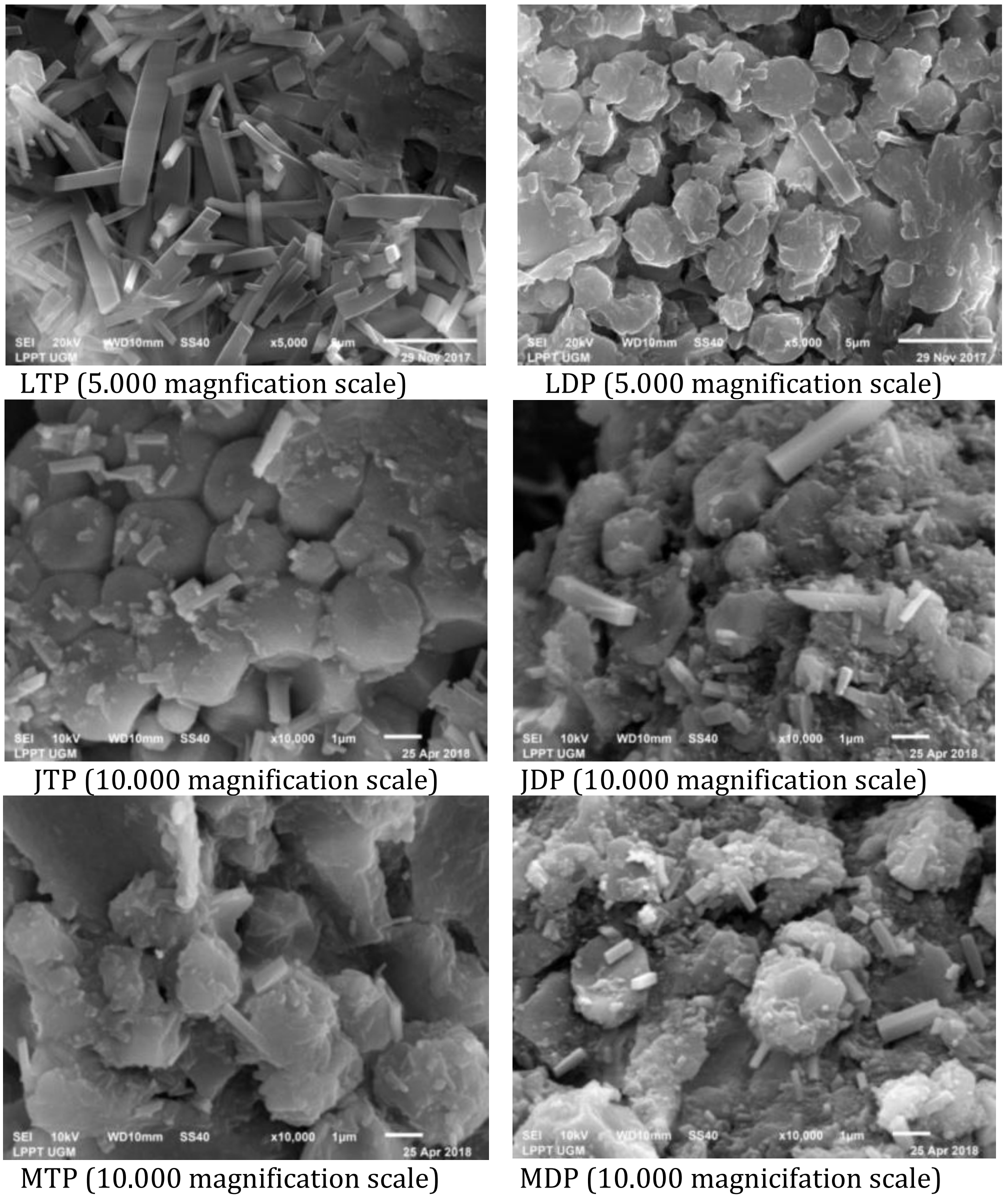


Figure 1 Observation results of Scanning Electron Microscope (SEM) on white pepper powder.

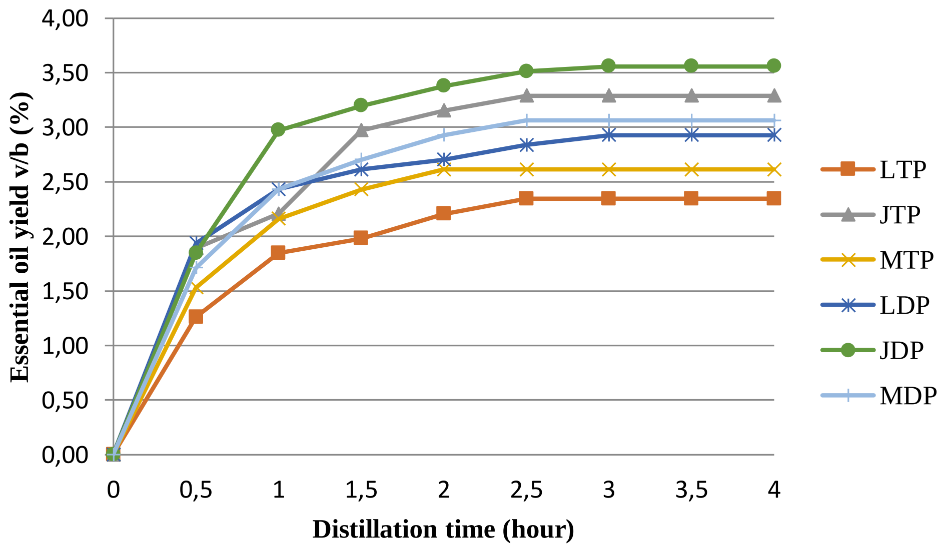


Figure 2. The effect of distillation time and microwave treatment on the white pepper essential oil yield from 3 (three) accesions without and with microwave treatment.

Table 2. Profile of volatile compounds white pepper essential oil from 3 (three) accesions without and with microwave treatment

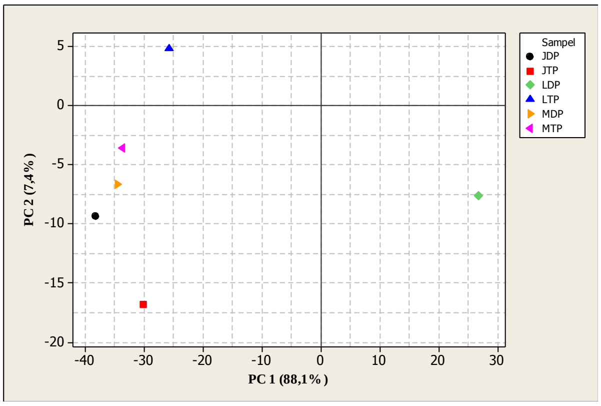
No.	Compound	Molekular formula	Retention Time (min)	LTP (%)	LDP (%)	JTP (%)	JDP (%)	MTP (%)	MDP (%)
1.	α-pinene	$C_{10}H_{16}$	3.51	3.88	14.14	7.47	-	3.78	3.85
2.	Camphene	$C_{10}H_{16}$	3.69	0.07	0.22	0.25	0.29	0.07	0.07
3.	β-phellandrene	$C_{10}H_{16}$	3.96	0.13	0.76	0.25	0.38	0.12	0.13
4.	β-pinene	$C_{10}H_{16}$	4.03	8.32	19.29	11.43	6.84	9.17	9.16
5.	β-myrcene	$C_{10}H_{16}$	4.12	2.89	-	2.66	-	2.53	2.74
6.	2-Pyridinepropanoic acid	$C_{11}H_{13}NO_3$	4.23	-	1.64	-	-	1.02	0.39
7.	1-phellandrene	$C_{10}H_{16}$	4.35	4.59	0.19	1.74	0.02	3.97	4.15
8.	δ-3-carene	$C_{10}H_{16}$	4.42	-	-	7.22	9.29	-	-
9.	β-ocimene	$C_{10}H_{16}$	4.44	17.42	-	-	10.97	11.59	8.45
10.	α-terpinene	$C_{10}H_{16}$	4.49	-	-	0.12	0.18	-	-
11.	Benzene	$C_{10}H_{14}$	4.60	1	0.15	0.4	0.7	-	-
12.	Limonene	$C_{10}H_{16}$	4.67	11.26	18.67	7.64	9.47	8.67	5.74
13.	Cyclohexene	$C_{10}H_{16}$	4.87	-	-	0.15	0.17	0.32	1.05
14.	α-thujene	$C_{10}H_{16}$	5.02	0.09	16.08	-	1.84	0.2	0.12
15.	γ-terpinene	$C_{10}H_{16}$	5.06	0.2	28.85	-	0.08	0.21	0.18
16.	α-terpinolene	$C_{10}H_{16}$	5.44	0.46	-	0.54	0.56	0.81	0.37
17.	δ-4-carene	$C_{10}H_{16}$	5.48	1.18	-	0.13	0.13	-	-
18.	Linalool	$C_{10}H_{18}O$	5.58	0.37	-	0.38	0.39	0.3	0.38
19.	β-penchylalcohol	$C_{10}H_{18}$	6.95	0.22	-	0.19	0.18	0.09	0.12
20.	Sabinene hydrat	$C_{10}H_{18}O$	7.28	0.07	-	-	-	-	-
21.	δ-elemene	$C_{15}H_{24}$	9.09	3.92	-	5.95	7.08	4.11	5.67
22.	α-cubebene	$C_{15}H_{24}$	9.26	0.08	-	-	-	-	-
23.	α-copaene	$C_{15}H_{24}$	9.66	2.4	-	0.14	0.12	1.14	1.67
24.	Valencene	$C_{15}H_{24}$	9.84	0.7	-	0.13	-	-	-
25.	β-caryophyllene	$C_{15}H_{24}$	10.33	34.73	-	43.81	50.51	46.02	46.71
26.	α-guaiene	$C_{15}H_{24}$	10.49	-	-	0.78	-	-	-
27.	α-humulene	$C_{15}H_{24}$	10.74	2.36	-	3.52	-	2.32	3.16
28.	Germacrene B	$C_{15}H_{24}$	10.90	-	-	-	-	0.07	0.12
29.	β-elemene	$C_{15}H_{24}$	10.91	0.08	-	0.86	0.79	-	0.71
30.	Germacrene D	$C_{15}H_{24}$	11.08	-	-	-	-	0.07	0.11
31.	Germacrene	$C_{15}H_{24}$	11.09	0.13	-	0.22	-	0.45	-
32.	β-selinene	$C_{15}H_{24}$	11.18	0.17	-	0.82	-	0.1	0.16
33.	α-selinene	$C_{15}H_{24}$	11.29	0.18	-	0.56	-	-	0.18
34.	δ-cadinene	$C_{15}H_{24}$	11.59	0.99	-	-	-	0.43	0.68
35.	γ-caryophyllene	$C_{15}H_{24}$	12.32	-	-	0.1	-	-	-
36.	Caryophyllene oxide	$C_{15}H_{24}O$	12.43	0.92	-	1.27	-	1.07	1.58
37.	β-guaiene	$C_{15}H_{24}$	12.85	-	-	-	-	-	0.07
38.	Pentalene	$C_{15}H_{24}O$	12.94	1.06	-	0.8	-	0.95	1.62
39.	Longipinocarveol	$C_{15}H_{24}O$	13.06	-	-	0.34	-	0.35	0.54
40.	β-copaene	$C_{15}H_{24}O$	13.07	0.13	-	-	-	-	-
41.	δ-guaiene	$C_{15}H_{24}$	13.27	-	-	0.13	-	-	-
42.	Spathulenol	$C_{15}H_{24}O$	13.45	-	-	-	-	0.09	0.11
	Total			100	99.99	100	99.99	100	99.99
	Monoterpene ($C_{10}H_{18}$)			50.49	98.2	39.60	40.42	41.44	36.03
	Sisquiterpene ($C_{15}H_{24}$)			45.74	0	57.02	58.31	54.69	59.23
	Oxygenated terpene			2.55	-	2.79	0.39	2.76	4.23
	Others			1.22	1.79	0.59	0.88	1.11	0.51
	M/S			1.10	∞	0.69	0.69	0.76	0.61

notes : - = not detected; M/S = the ratio of monoterpenes to sisquiterpenes.

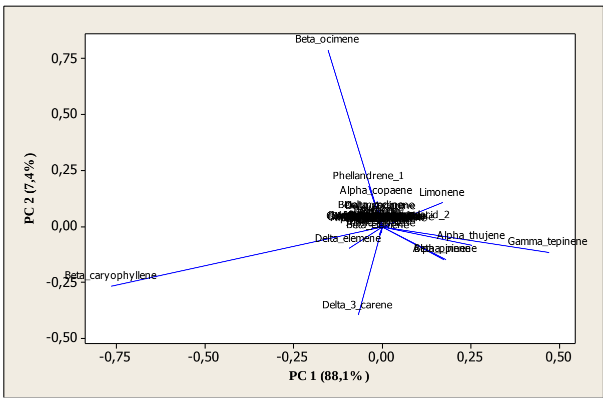
Profile of volatile compounds in white pepper essential oils

There were 42 volatile compounds identified by GC-MS (Table 2). The number of compounds identified in LTP was 30 compounds while in LDP was 10 compounds. There were 30 compounds identified in JTP, while in JDP were 20 compounds. The compounds identified in MTP were 28, while in MDP were 30. The major component of white pepper essential oil in this study was β-caryophyllene (50.51%).

Based on the score plot, there was a difference between the profile of volatile compounds from Lampung daun lebar accession and that of two other accessions (Figure 3). The Score plot showed that LTP and LDP point positions were in different quadrants, while JTP, JDP, MTP, and MDP were in the same quadrant. The loading plot showed the main component in LTP was determined by the β-ocimene compound while LDP was by γ-terpinene. In JTP, JDP, MTP, and MDP, the main components were determined by the β-caryophyllene compound.



(a)



(b)

Figure 3. Score plot (a) and loading plot (b) PCA profil of volatile compounds white pepper essential oil from 3 (three) accesions without and with microwave treatment.

Characteristics of White Pepper Essential Oils

The results of the analysis showed that different pepper accession produced different L*, a*, and b* values (Table 3). The microwave treatment caused changes in the values of b* but did not affect the values of L* and a*. Based on Table 3, microwave treatment caused increases in the values of b*, making the color of the white pepper essential oils bluer. The value of b* increased 44.57% in Lampung daun lebar, 73.40% in Jambi, and 201.99% in Merapin jumbo. The highest b*value (- 9.12) was found in Merapin jumbo accession with microwave treatment, making the color of the essential oil blue.

Table 3. The value of L *, a *, and b * white pepper essential oil from 3 (three) accesions without and with microwave treatment.

Sample	L*	a*	b*
LTP	$58.74^{b} \pm 0.06$	$2.39^{bc} \pm 0.07$	$-2.19^{d} \pm 0.02$
LDP	$59.16^{d} \pm 0.06$	$2.44^{bc} \pm 0.06$	$-3.17^{b} \pm 0.12$
JTP	$59.09^{cd} \pm 0.00$	$2.39^{bc} \pm 0.01$	$-1.88^{e} \pm 0.09$
JDP	$59.21^{d} \pm 0.34$	$2.56^{c} \pm 0.01$	$-3.26^{b} \pm 0.00$
MTP	$58.83^{bc} \pm 0.09$	$2.29^{b} \pm 0.02$	$-3.02^{c} \pm 0.10$
MDP	$58.38^{a} \pm 0.17$	$1.90^{a} \pm 0.02$	$-9.12^{a} \pm 0.04$

notes: different letters in the same column show significant differences ($p<0.05$).

Different pepper accession produced different specific gravity, solubility in 95% ethanol, and acid number (Table 4). The microwave treatment increased the specific gravity but reduced solubility in 95% ethanol and the acid number of white pepper essential oil. For Lampung daun lebar, specific gravity increased by 1.17%, solubility in 95% ethanol decreased by 17.95%, and the acid number decreased by 20.8%. For Jambi accession, specific gravity increased by 1.27%, solubility in ethanol 95% decreased by 17.14%, and the acid number decreased by 20.51%. Specific gravity in Merapin Jumbo increased by 1.04%, solubility in ethanol 95% decreased by 15.79%, and the acid number decreased by 16.8%. Friedman test produced a chi-square value of 3.732 ($p>0.05$) so that it can be concluded that pepper accessions and microwave treatment did not affect the refractive index of white pepper essential oils.

Antioxidant Activity of White Pepper Essential Oils

Different pepper accession produced different antioxidant activities (Table 5). The interaction between the accessions of pepper and the microwave treatment affected the antioxidant activity. Microwave treatment increases antioxidant activities in Lampung daun lebar accession by 7.4%, Jambi by 5.15%, and Merapin jumbo by 5.88%. The highest antioxidant activity was 74.95% in the Merapin jumbo accession with microwave treatment, while the lowest was in the

Jambi accession without microwave treatment, which is 52.59%.

Table 5. Specific gravity, solubility in 95% ethanol and acid number white pepper essential oil from 3 (three) accesions without and with microwave treatment.

Sample	Specific gravity (b/b)	Solubility in 95% ethanol (v/v)	Acid Number
LTP	$0.856^{a} \pm 0.002$	$0.39^{c} \pm 0.02$	$1.25^{d} \pm 0.05$
LDP	$0.866^{c} \pm 0.001$	$0.32^{a} \pm 0.01$	$0.99^{bc} \pm 0.05$
JTP	$0.867^{c} \pm 0.001$	$0.35^{b} \pm 0.02$	$1.17^{d} \pm 0.05$
JDP	$0.878^{e} \pm 0.002$	$0.29^{a} \pm 0.01$	$0.93^{a} \pm 0.05$
MTP	$0.863^{b} \pm 0.001$	$0.38^{bc} \pm 0.02$	$1.25^{d} \pm 0.05$
MDP	$0.872^{d} \pm 0.001$	$0.32^{a} \pm 0.01$	$1.04^{c} \pm 0.08$

notes: different letters in the same column show significant differences ($p<0.05$).

Table 5. Antioxidant activity (%) white pepper essential oil from 3 (three) accesions without and with microwave treatment

Sample	Antioxidant Activity (%)
LTP	$63.93^{c} \pm 0.11$
LDP	$68.66^{d} \pm 0,10$
JTP	$52.59^{a} \pm 0.23$
JDP	$55.30^{b} \pm 0.21$
MTP	$70.79^{e} \pm 0.00$
MDP	$74.95^{f} \pm 0.12$

notes: different letters in the same column show significant differences ($p<0.05$).

4. Discussion

Armstrong (1999) reported that water has a higher degree of dielectric constant in microwave absorption so that the rate of absorption of microwave energy in water is higher than the rate of heat dissipation in the system. This causes the superheating effect to occur when water is inside a matrix. This condition allows more evaporation of water (Uquiche *et al.* 2008). The water content of white pepper without microwave treatment as shown in Table 1 is below 14% and still meets the quality standards required by SNI 0004: 2013 (National Standardization Agency 2013). In general, the water content of white pepper dried using sunlight is in the range of 12-14%, and most of the water lost during drying is the surface water in the white pepper. Therefore, the water content of white pepper treated with microwave is below 12% due to the evaporation of residual surface water and free water outside and inside the white pepper cell. For Lampung daun lebar and Jambi accessions, microwave treatment caused the piperine content to decrease, while for Merapin jumbo accession, it raised piperine content equal to 10.57%. This result conforms with what reported by Syafrudin (2016), that the more water loses in the material, the more effective will be the piperine extraction process by the solvent resulting in higher piperine content.

Syafrudin (2016) and Lumbessy (2016) reported that microwave treatment causes cells to suffer from form damage or merged. Preliminary microwave treatment causes damage to white pepper cells, especially cellulose, in the cell wall that envelops the essential oil glands. This was confirmed with the SEM observations, showing that the white pepper treated with microwave experienced more cell tissue damage than those that were not treated with a microwave. Microwave treatment caused modification of the cellular walls, resulting in greater porosity. According to Uquiche *et al.* (2008), the penetration of energy produced by microwave can degrade cellulose molecules that envelop white pepper cell tissue and cause cell wall permeability to decrease, eventually causing the cell to become intact.

Microwave treatment causes a decrease in water content and degrades the cellulose structure of white pepper cell walls. This will have a positive effect on the effectiveness and efficiency of the distillation process of white pepper essential oil so that it can increase the yield of essential oils obtained. Based on Figure 2, the essential oils obtained after distillation lasts for half an hour varied, where the lowest yield of essential oil was in Lampung Daun Lebar without microwave treatment (LTP) and the highest was in with microwave treatment (LDP). The distillation time of half an hour was the first phase where the essential oils obtained were dominated by the rupture of the vacuole containing the essential oil cells due to the size reduction process. This conforms with what was reported by Orav *et al.* (2004), that the yield obtained from pepper powder is higher than that of white peppercorn. The second phase began after the distillation lasts for one hour, where the yield of essential oils obtained increasing as the result of raising the temperature in the system. Essential oils are composed of components with high volatility. The more the damaged cell walls surrounding the essential oils are, the faster and the more essential oils come out of the material, then this condition will be maximum when the distillation time reaches 2.5-3 hours. This result conforms with what was reported by Uquiche *et al.* (2008), that microwave heating vaporizes the water of the sample substrate microstructure, increasing the pressure in its interior; realizing water from the microwave heating causes the disintegration of the material, cell membrane rupture, and improving the efficiency of the pressing extraction of essential oil from the sample, enabling the passage of oil from the cell membrane. The third phase started when there was no addition of the essential oil obtained and after reaching a processing time of 4 hours, the distillation was stopped.

The results of the analysis showed that different pepper accessions produced different yields of essential oils. Jeevitha *et al.* (2016), reported that pepper given microwave treatment prior to distillation had a higher essential oil yield than without microwave treatment. The water content in white pepper before being distilled can affect the yield of essential oil obtained. The lower the water content of white pepper is, the higher the essential oil yield will be. The microwave treatment with the correct duration and the use of power is able to maximize the damage to the white pepper cell network so that what is evaporated by microwave energy is dominated by water and minimizes the loss of volatile components.

The number of compounds identified in this study was 42, more than what was reported by Abd El Mageed *et al.* (2011) totaling 26 compounds, Fan *et al.* (2020) totaling 40 compounds, and Liu *et al.* (2013) totaling 41 compounds. The difference in the number of these compounds is due to the different accessions of pepper used as raw material and the different geographical conditions of the origin. It is presumed that the decrease in the number of volatile compounds in the Lampung daun lebar and Jambi accessions given the microwave treatment was caused by decomposition and resinification. Therefore, further research is needed to determine whether different duration and power of microwave treatment are required for different accessions. Molecular movements and heat generated during the microwave treatment can cause volatile compounds from the sesquiterpenes to decompose and form simpler compounds, which are monoterpenes. Therefore, the percentage of monoterpenes increases in line with the absence of the sesquiterpene class. Related to the occurrence of resinification, according to Jeevitha *et al.* (2016), the microwave treatment raise the temperature forming more resin. Resinification of the sesquiterpene class into resin potentially causes this class to be undetectable when the essential oil of white pepper is analyzed using GC-MS, since the resin generally settles, making it missed when the sample is taken out of the sampling bottle.

This study shows that white pepper essential oils have higher volatile compounds from the sesquiterpene class than from the monoterpene class and this result conforms with was reported by Wang *et al.* (2018). Pepper essential oil with a high sesquiterpene composition produces an aroma similar to that of pepper. Abd El Mageed *et al.* (2011) stated that the presence of sesquiterpene compounds in large quantities is desired because they are responsible for the flavor of the pepper. In this study, off-odorants such as β-damascenone, eugenol, skatole, guaiacol and piperonal were not found. According to Putri *et al.* (2018), the quality of white pepper is also influenced by the soaking and post-washing processes, in that this study also shows the absence of off-odorants in the sample used is the result of good processing and correct storing method. The good process requires the pepper to be soaked in water for 7-14 days, washed in running water, and dried in a place away from potential contaminants. The right method to store the white pepper is by sorting it before the storage and packing it in good packaging to avoid direct contact with factors that can degrade the quality of white pepper.

Based on Figure 3, 2 (two) principal components selected from the 2 (two) eigenvectors with the highest eigenvalues can cover more than 50% of the total data variations. The effect of the principal component on the variation of the first line data (PC1) was 88.1% and the second line principal component had an effect of 7.4% (PC2). This study shows that Principal Component Analysis (PCA) can classify samples based on their constituent compounds and produce three sample groups. The analysis can be used to facilitate the interpretation of GC-MS results. This result is supported by Piggott & Othman (1993) in that PCA can distinguish essential oils of 3 (three) pepper varieties with and without irradiation treatments.

In samples treated with microwave, the increase in specific gravity shows that the number of sesquiterpenes was more than monoterpenes. The decrease in solubility in ethanol 95% shows that the number of terpenes was more than oxygenated terpenes, and the decrease in acid numbers shows that fewer oxidation reactions occurred. These results indicate that the microwave treatment causes the distillation process to take place more effectively and efficiently, so that it does not cause significant changes in the characteristics of the essential oils of white pepper and the quality of the essential oils does not decrease. The results of this study show that the characteristic of white pepper essential oils, which include color, specific gravity, solubility in ethanol 95%, and the acid number still meet Indonesian pepper essential oil quality standards in ISO 3061:2008, and these results are not much different from those reported by Anggraini *et al.* (2018).

These results are consistent with those reported by Abd El Mageed *et al.* (2011), in that antioxidant activities on white pepper essential oils given heating treatment using a microwave oven were higher than without microwave oven heating and given conventional heating treatment. Gülçin (2005) reported that antioxidant activity in pepper essential oils is due to its strong ability to donate hydrogen atoms. That ability is expected to be stronger when the pepper is given a microwave treatment before distillation.

5. Conclusion

Different pepper accessions produced different profiles of volatile compounds, characteristics, and antioxidant activities. Microwave treatment was conducted to damage the cell walls of white pepper, lower water content, raise piperin content and essential oil yield, and cause the change in the profile of volatile compounds of white pepper essential oil in Lampung daun lebar accession. The treatment made the color of white pepper essential oils bluer, raised the specific gravity, lowered the solubility in ethanol 95% and the acid number, and raised antioxidant activities, while the refractive index was not affected by pepper accessions and the microwave treatment. These results suggest that microwave treatment can effectively increase the white pepper essential oil yield without a significant loss in the product quality.

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7. Declaration of Conflicting Interests

The authors have declared no potential conflicts of interest concerning the study, authorship, and/or publication of this article.

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